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(54) METHOD OF MAKING A DUO-DENSITY SILICON NITRIDE ARTICLE

(71) We, FORD MOTOR COMPANY LIMITED, of Eagle Way, Brentwood, Essex CM13 3BW, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of making a duo-density article of silicon nitride.

Silicon nitride has a wide variety of uses based on its physical and chemical properties. These uses, to name a few, include thermocouple protection tubes, crucibles for foundry use, substrates for electronic applications and structural components for gas turbine engines.

Silicon nitride can be produced by a number of different processing techniques with each technique yielding a different final density. Each technique also has a definite restriction on the final shape which can be produced. Simple shapes of better than 98% of theoretical density can be made by hot pressing silicon nitride powder to form the final article. Complex shapes, however, generally cannot be manufactured by this processing technique.

As an alternate to the hot pressing techniques, silicon nitride articles of complex shape having densities of 70 to 75 percent of theoretical density are produced by an injection molding technique. In this technique, silicon metal particles and a thermoplastic are formed into a mixture. This mixture is injection molded to form the shape of the article. Subsequent operations include the heating of the article to burn out the thermoplastic and the nitriding operation to produce the final silicon nitride article.

Another processing technique for manufacturing fairly intricate shapes of silicon nitride is slip casting. A slip casting technique generally produces a final article having a density in the range from 80—85% of theoretical density. In the slip casting technique, silicon metal particles are cast into the desired shape. The silicon metal is then converted in a nitriding operation, into silicon nitride.

It is generally impossible to fabricate a complete rotor for a gas turbine engine of hot pressed silicon nitride material. The impossibility of manufacturing such a complete rotor by a hot pressing technique comes about because of the complex shape of the rotor blades. The complex shape of such blades can be formed easily by either an injection molding technique or a slip casting technique. It is generally impossible, however, to form a complete rotor by an injection molding technique or slip casting technique as the hub portion of the rotor formed by such a technique cannot withstand the mechanical and thermal stresses imposed upon that portion of the rotor during its use in an engine.

This invention teaches a method wherein the best characteristics of a hot pressed silicon nitride material are used in conjunction with either an injection molded or a slip cast silicon nitride material to form a complex article of manufacture such as a rotor for a gas turbine engine.

The method of this invention has the following steps. A first element is formed of silicon metal particles in a silicon metal forming operation. The first element is nitrided so that the silicon metal particles forming the first element are converted to substantially pure silicon nitride. All but a bonding surface of the first element is coated with a release agent and all but the bonding surface of the first element are encapsulated with silicon nitride thereby to form a die member. The die member is placed in a pressing die structure so that the bonding surface of the die member forms a portion of the total die surface defining the interior surface of a die volume formed within the pressing die structure. The die volume is filled with a mixture consisting of from 94 to 99.5% by weight silicon nitride particles and from 6.0 to 0.5% by weight of a densification aid. The mixture is hot-pressed under the simultaneous application of heat and pressure in the die volume to at least

98% of theoretical density thereby forming a second element of the duo density article while simultaneously bonding a surface area of the second element to the first element along the first element's bonding surface. The encapsulant formed about the first element is removed to produce the final article of silicon nitride having two zones with different densities.

The first element of silicon metal particles may be formed by either a slip casting operation or an injection molding operation. The encapsulation of the first element with silicon nitride may be accomplished by coating the first element with a material which forms a thin protective barrier and then by injection molding or slip casting silicon particles therearound, the silicon particles subsequently being nitrided to form an encapsulating material. The encapsulant may be applied in more than one step so as to ease the removal of the encapsulant material.

The invention will now be described with reference to the accompanying drawings in which:—

Figure 1 is a schematic drawing, in cross-section showing apparatus capable of performing the method of this invention. Figures 2, 3 and 4 illustrate graphically the manner in which a first element of silicon nitride can be encapsulated.

A process for making a duo-density article of silicon nitride will be described. The article to be described will be a rotor for a gas turbine engine. The first element of the rotor is its outer blade ring of complex shape over which the hot gases of the engine flow to turn the rotor.

The method of making a duo-density article of silicon nitride in accordance with the teachings of this invention is initiated by forming the first element, which in this case, is the blade ring of the rotor. The first element may be formed by any generally known forming technique. Illustrative types of forming techniques are slip casting and injection molding.

In the case of forming the first element by a slip casting technique, silicon metal particles, suspended in a vehicle, are cast into a mold having the shape desired for the blade ring. The mold is positioned against a porous material capable of drawing the vehicle of the slip out of the casting volume thereby leaving behind a consolidated mass of silicon metal particles. In such a manner the first element is formed of silicon metal particles by a slip casting technique.

As an alternative approach, the general shape of the first element may be formed by an injection molding technique, a typical injection molding compound for forming the first element is one which would have about 60 to 66 percent by volume of silicon metal particles with the remainder being a thermoplastic binder. In general, the particle size of the metal will be such that the material will have a maximum particle size in the range of 40 to 60 microns and a mean particle size in a range of from 10 to 13 microns. Once a molding composition is formed, the composition is fed into a cylinder of an injection molding machine. The machine heats the thermoplastic above its melting point. Pressure is applied to the cylinder and the molding composition is shot into a cold molding die having the configuration of the blade ring to be produced. The thermoplastic solidifies into the desired shape thereby locking the silicon metal particles carried along with it into the desired shape.

The so formed blade ring of silicon metal particles and thermoplastic is gradually heated in a furnace to a temperature of about 350°C. The heating program may take as long as three days so that no stresses are created in the article during heating. During this heating, the thermoplastic binder is burned out. This action leaves behind silicon metal particles in the desired shape of the blade ring.

No matter how the silicon metal particles are formed into the blade ring shape desired, the so formed first element is thereafter nitrided in order to produce a finished body of silicon nitride. Since the first element is formed of silicon metal particles, the nitriding operation is effective to change the first element into a silicon nitride. In the nitriding operation the element is heated while exposed to nitrogen gas at a temperature and for a sufficient period of time that all of the silicon is transformed into silicon nitride. A full procedure for nitriding silicon to form silicon nitride is disclosed in our United Kingdom Patent Specification No. 1,448,915. If the article nitrided had been originally formed by an injection molding process, the final article will have a density of 70 to 75 percent of theoretical density. If the original article has been formed by a slip casting technique, the finished article will have a density in the range of 80 to 85 percent of theoretical density. Upon completion of the nitriding step, the finished first element is a blade ring 10 (Figure 2) having a plurality of blades 12 thereon attached to a support portion 14. The support portion 14 also defines an inwardly facing bonding surface 16, best seen in Figure 1.

The method of this invention teaches the formation and simultaneous bonding of a second element in this case a hub, to the bonding surface 16 of the first element. The hub serves as a support for the blade ring 10. In order to carry out the bonding operation it is necessary to encapsulate the blade ring so that it is capable of withstanding the pressure encountered in a hot press

forming and bonding operation. In order to carry out the encapsulation, the steps shown generally in Figures 2, 3 and 4 are followed.

One procedure for encapsulating the blade ring 10 is to coat the blade ring in its entirety with a thin coat of boron nitride powder to serve as a release material for subsequent ease in removing the encapsulant. The blade ring is then placed in a mold which closes off the volume between each of the blades thereby defining a plurality of individual cavities. A casting slip of silicon metal particles is supplied to each of the cavities to build up slip cast silicon material. After the slip casting operation, the material now in place between the blades is nitrided to produce individual silicon nitride support portions 18, see Figure 3, between each of the blades 12.

After placing the support portions 18 between each of the blades 12, the article is once again coated with boron nitride powder. The article is placed in a second forming mold and a silicon metal slip is cast around the entire volume of the blade ring 10 with the exception of the bonding surface 16. After the slip casting operation, the slip cast silicon particles are nitrided to produce a silicon nitride body 20 as an encapsulant. Thus, after this operation the silicon nitride body 20 contains therewithin the blades 12 and leaves exposed only the bonding surface 16 thereof. The body 20 is sized so as to be receivable in a pressing die structure shown in Figure 1.

The pressing die structure 22 includes a contoured bottom graphite piston 24, tapered graphite inside wedges 26, tapered graphite outside wedges 28, a graphite restraining sleeve 30, a contoured top graphite piston 32, an outside graphite piston 34 for applying pressure to the wedges 28, an outside graphite sleeve 36, an inside cooling ram 38 and an outside cooling ram 40.

After the body 20 is formed with only the bonding surface 16 of the blade ring 10 being exposed, the body is placed on the contoured bottom graphite piston 24 as shown in Figure 1. At that time, the various graphite wedges and restraining sleeves are positioned about the body 20. A measured volume of a silicon nitride powder suitable for a hot pressing operation is poured into a volume defined between surfaces of the contoured bottom graphite piston 24, the bonding surface 16 and the graphite restraining sleeve 30. The contoured top graphite piston 32 is then placed between the graphite restraining sleeve and pressure is applied to the material in the defined volume.

The powder used in the pressing operation is a mixture consisting of from 94 to 99.5% by weight of silicon nitride particles and from 6.0 to 0.5 percent by weight of a densification aid. In general, the silicon nitride used in the compacting operation is alpha silicon nitride powder. The powder is generally a ceramic grade and is preferably all minus 325 mesh (US and ASTM standard sieve No). The silicon nitride powder is wet ball milled in a rubber lined mill with alumina or tungsten carbide balls and alcohol for a time ranging from one day to two weeks. A densification aid, such as magnesium oxide or any other suitable material, is mixed thoroughly with the silicon nitride powder during the milling operation. Concentrations of densification aid are in the range of from 0.5 to 6.0 weight percent. The aid helps in the powder compaction process. After milling, the silicon nitride powder slurry is dried and screened through a 100 mesh (US and ASTM standard sieve No.) screen in preparation for a hot pressing operation.

As stated above, the silicon nitride powder 42 is placed in the volume defined between the various die members in order to initiate a pressing operation. A measured amount of the material is placed therein so as to produce a final article of a particular size. The material will be hot pressed to form a second element which in this case is a hub 44 for the blade ring 10. The hub will be formed and simultaneously bonded to the blade ring during this processing and the hub will exhibit its final dimensions and contours. A barrier material can be coated on the graphite die system to minimize any reaction between the silicon nitride powder and the die system. Barrier materials commonly used are graphite foil and boron nitride. The materials would be placed on all of the surface of the interior volume of the pressing die structure except the bonding surface 16 of the blade ring 10.

The silicon nitride material 42 is hot pressed at a temperature in a range from about 1650°C to about 1800°C and a pressure from about 2,000 psi to about 4,000 psi. The heating of the material is accomplished by an induction heating unit, not shown. The pressure, of course, is applied by applying a compressive force or the contoured bottom piston 24 and the inside cooling ram 38 thereby compacting the silicon nitride material. The final hot pressed hub 44 will have a density of 98 percent theoretical or greater. The silicon nitride hub 44 will readily withstand both the temperatures and the stress conditions imposed upon it when used as the hub portion of a rotor in a gas turbine engine.

After the pressing operation the pressing apparatus is turned off and allowed to cool slowly back to room temperature. The press bonded rotor assembly is allowed to cool simply by leaving it in ambient conditions. When the assembly is cooled, the removable graphite die elements are removed to take

the now almost finished turbine rotor therefrom. The one thing left to finish on the article is to remove the encapsulant from the blade ring 10. As previously mentioned, the blade ring had been treated with a release agent such as boron nitride to help in its releasing the encapsulant from its surfaces. It has been found that if the finished article is immersed in a bath to which ultra sonic energy is applied, the barrier material along the planes of association will loosen and open up which allows easy removal of the individual support portions between the blades.

The so finished article is thus one which has a silicon nitride hub of near theoretical density and a complex blade ring of a different density. The finished article can be used as a rotor for a gas turbine engine.

WHAT WE CLAIM IS:—

1. A method of making a duo-density article of silicon nitride which comprises the steps of:

making a first element of silicon metal particles by forming silicon metal particles in a forming operation;

nitriding said first element so that said silicon metal particles forming said first element are converted to substantially pure silicon nitride;

coating all but a bonding surface of said first element with a release agent and then encapsulating all but said bonding surface of said first element with silicon nitride to form a die member;

placing said die member in a pressing die structure so that said bonding surface of said die member forms a portion of the total die surface defining the interior surface of a die volume formed by said pressing die structure;

filling said die volume of said pressing die structure with a mixture consisting of from 94 to 99.5% by weight silicon nitride particles and from 6.0 to 0.5% by weight of a densification aid;

compacting said mixture in said die volume under the simultaneous application of heat and pressure to at least 98% of theoretical density thereby forming a second element of the duo density article while simultaneously bonding said second element to said first element along said first element's bonding surface; and

removing said encapsulant about said first element to produce the final article of silicon nitride having two zones of different density.

2. The method of Claim 1 wherein: said first element of silicon metal particles is made by slip casting operation.

3. The method of Claim 1 wherein: said first element of silicon metal particles is made by an injection molding process.

4. The method of Claim 1 wherein: said encapsulating of all but said bonding surface of said first element is carried out by one or more repetitions of a process wherein silicon particles are slip cast on said element and subsequently nitrided, said release agent being applied to the surfaces to be encapsulated prior to each repetition of the encapsulating process.

5. The method of Claim 4 wherein: said release agent is boron nitride.

6. A method of making a duo-density article of silicon nitride substantially as hereinbefore described with reference to and as shown in the accompanying drawings.

7. An article made by the method claimed in any one of the preceding claims.

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1470949 COMPLETE SPECIFICATION

1 SHEET This drawing is a reproduction of the Original on a reduced scale

FIG.1

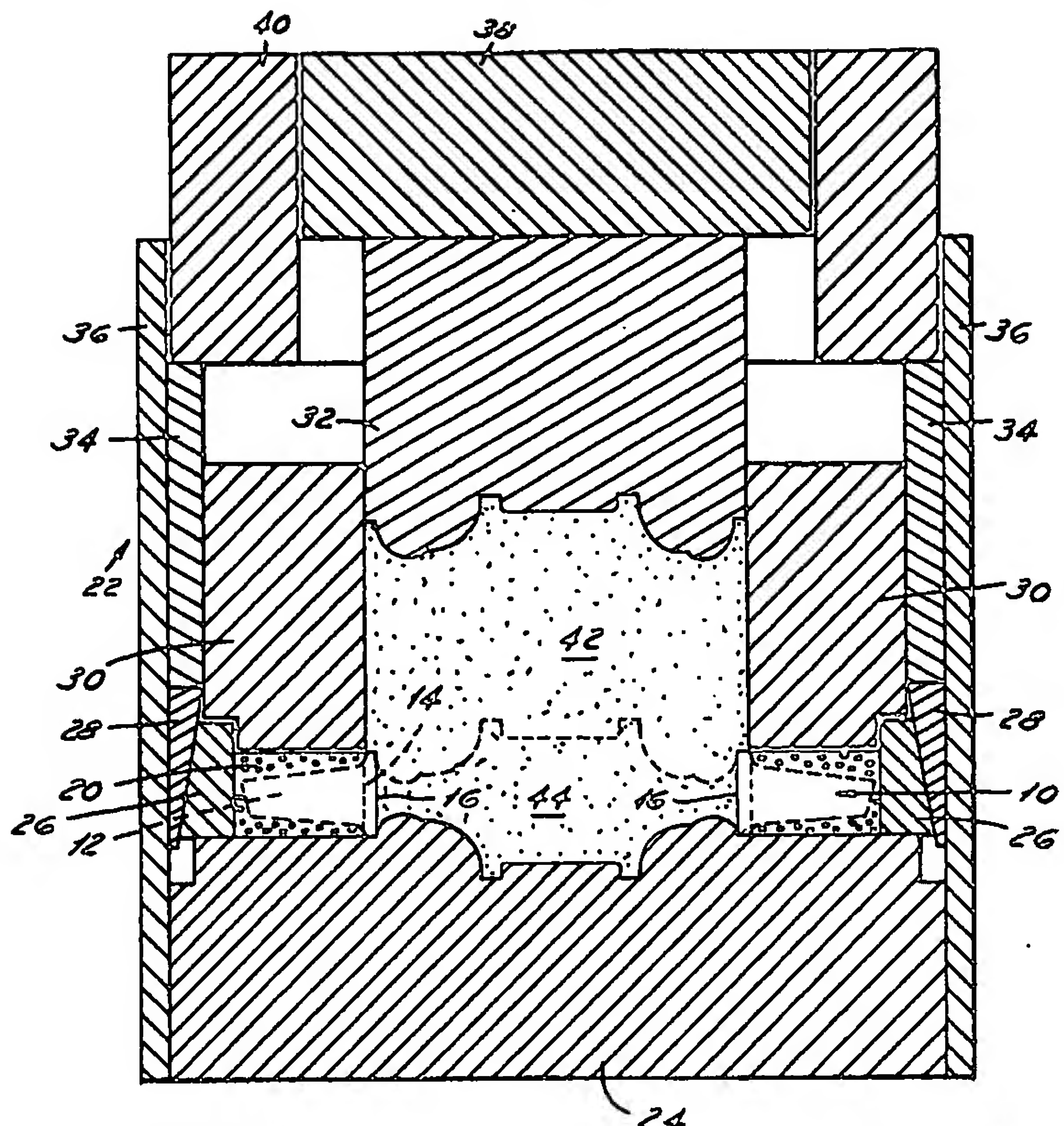

FIG.2

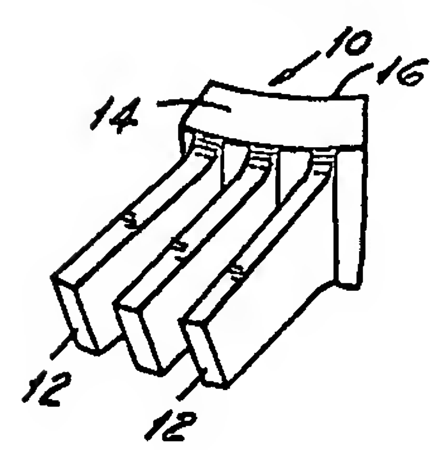

FIG.3

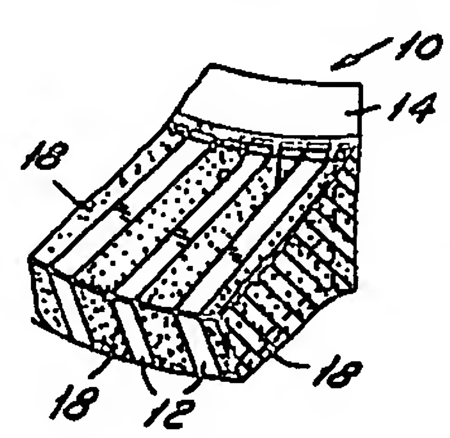

FIG.4